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(58) Field of search
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(54) Preparation of solid particulate components for detergents

(57) A process for the preparation of a solid particulate material suitable for use as a detergent composition or a component thereof comprises fluidising a particulate material, preferably an alkaline builder salt, in an internal recirculating fluidised bed and spraying thereon a liquid detergent active material or precursor therefor. In a preferred embodiment, the particulate fluidised material is sodium carbonate and the liquid sprayed on is an alkylbenzene sulphonic acid which is thereby neutralised *insitu*. A product containing 5-40% by weight of alkylbenzene sulphonate can be obtained.

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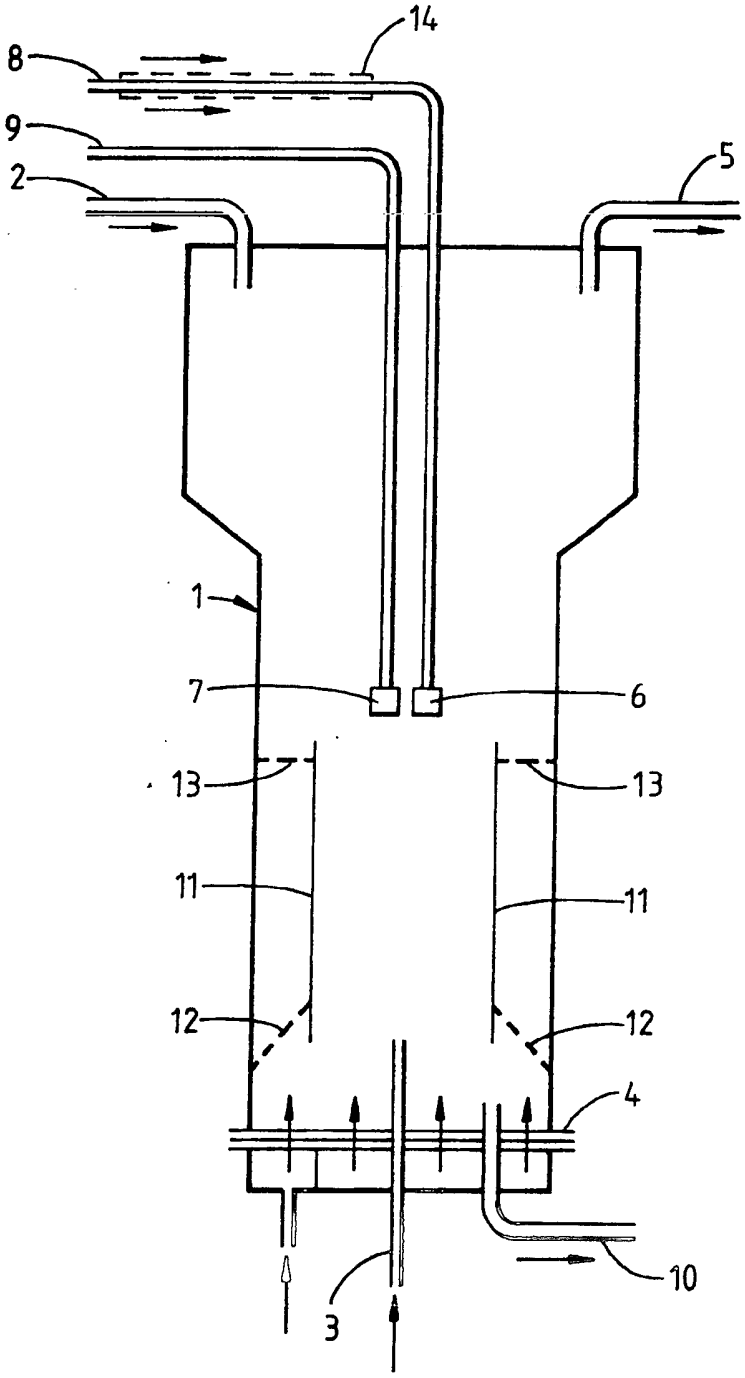
Fig.1



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PROCESS FOR THE PREPARATION OF PARTICULATE MATERIAL FOR DETERGENT COMPOSITIONS

FIELD OF INVENTION

The present invention is concerned with a process for the production of a particulate material containing at least one detergent-active compound. The particulate material is useful in its own right as a simple detergent composition, or as a component for a more sophisticated composition. The production process involves spraying the the detergent-active compound or a precursor thereof onto a particulate builder material in a fluidised bed.

BACKGROUND AND PRIOR ART

Detergent powders based on anionic detergent-active compounds are conventionally produced by spray-drying a slurry. A spray-drying tower, however, represents a substantial capital investment and may not be the most

efficient mode of operation in parts of the world, for example, developing countries, where distribution of product from a large central factory may be difficult or expensive. In such locations, there is a need for smaller, more flexible production units, and non-tower processes are of especial interest.

Also of increasing interest in recent years is the replacement of phosphate builders, said to cause eutrophication of lakes and rivers, by other materials, among which sodium carbonate is prominent.

East German Patent No. 140 987 (VEB Waschmittelwerk) discloses a continuous process for the production of granular washing and cleaning compositions, wherein liquid components such as nonionic surfactants or the acid precursors of anionic surfactants are sprayed onto a powdered builder material, especially sodium tripolyphosphate having a high phase II content.

The present inventors were interested in using a fluidised bed process for the production of detergent powders based on sodium carbonate, and attempted to spray alkylbenzene sulphonic acid onto soda ash in a conventional fluidised bed. It was not possible, however, to obtain a particulate free-flowing product by this method: except at very low alkylbenzene sulphonic acid loadings, only a lumpy, sticky product could be obtained. Unexpectedly, this problem was solved by the use of an internal recirculating fluidised bed rather than one of conventional design. An internal recirculating fluidised bed differs from a conventional fluidised bed in that it is provided with a central draught tube, which establishes desirable and predictable patterns of solids circulation and mixing; if a liquid is sprayed onto the bed material uniform and controlled liquid/solid contact occurs for a

finite time interval sufficient to allow the liquid to react with the solid bed material. The use of recirculating fluidised beds for the gasification of fossil fuels such as coal or fuel oil has been reported, but the use of these beds in the detergents industry has not hitherto been proposed.

DEFINITION OF THE INVENTION

The present invention provides a process for the preparation of a solid particulate material suitable for use as a detergent composition or a component thereof, which comprises fluidising a particulate material comprising at least one detergency builder salt in an internal recirculating fluidised bed, and spraying thereon liquid material comprising at least one detergent-active compound and/or at least one precursor thereof.

DESCRIPTION OF THE INVENTION

The process of the invention involves the preparation of a particulate detergent composition or component therefor by spraying at least one liquid ingredient onto a solid particulate base including at least one detergency builder salt, in a fluidised bed of a specific type, namely an internal recirculating fluidised bed.

The process is applicable to the production of any built particulate detergent composition, and the solids feed to the internal circulating fluidised bed may contain any of the conventional builder salts, and, if desired, other salts without a builder function, for example, sodium sulphate. In accordance with a preferred embodiment of the invention, described in more detail below, the particulate material comprises or consists of alkaline material capable of effecting the neutralisation

of a liquid acid precursor of an anionic detergent-active material. Examples of such alkaline materials, which also act as detergency builders, include alkali metal tripolyphosphate and, above all, alkali metal carbonate.

The liquid feed to the internal circulating fluidised bed may include any liquid or liquefiable ingredient which it is desired to incorporate in the final composition, but it is an essential feature of the invention that the sprayed-on liquid includes at least one detergent-active compound, or a precursor thereof that will be converted to the detergent-active form by reaction with the solid material of the fluidised bed. Anionic detergent-active compounds may conveniently be sprayed on in concentrated aqueous (for example, about 60-80% by weight) solution form. According to the preferred embodiment of the invention mentioned above, however, those anionic detergent-active compounds having a stable acid form, notably alkylbenzene sulphonates, are preferably incorporated in that form and react with the alkaline bed material to form the anionic detergent-active compound in situ. This method is not suitable for incorporating anionic detergent-active compounds of which the acid form is not stable, for example, alkyl sulphates, alkyl ether sulphates and olefin sulphonates.

If desired, other liquid ingredients may also be sprayed onto the fluidised bed. One material frequently used in detergent compositions that may conveniently be incorporated by this method is sodium silicate, in concentrated aqueous solution form.

The process of the invention may be carried out either batchwise or continuously.

PREFERRED EMBODIMENT OF THE INVENTION

As indicated previously, according to a preferred embodiment of the invention, the solid particulate material of the fluidised bed comprises one or more alkaline builder salts, and the liquid feed comprises at least one acid precursor of an anionic detergent-active compound.

The solid feed to the internal recirculating fluidised bed preferably includes, and more preferably consists wholly or predominantly of, alkali metal carbonate: the preferred material is sodium carbonate. This will generally be in anhydrous form, as soda ash. If the solid feed consists wholly of soda ash, the resulting product will be a mixture of sodium carbonate and the anionic detergent-active compound in sodium salt form: the amount of the anionic detergent-active compound in the product is preferably at least 5%, more preferably at least 15%, and may advantageously be above 20%, for example, 25-35%: the use of an internal recirculating fluidised bed in accordance with the invention allows substantially higher take up of the liquid acid than can be obtained using a conventional fluidised bed. If desired, other solid materials may be present depending on the desired composition of the final product; these need not necessarily be alkaline, provided that sufficient alkaline material is present both to neutralise the sprayed-on acid and to provide builder and buffer capability in the final product.

In this embodiment of the invention, the liquid feed to the internal recirculating fluidised bed comprises the acid form of an anionic detergent-active compound. The preferred material is an alkylbenzene sulphonic acid, preferably a linear alkylbenzene sulphonic acid containing

from 8 to 15 carbon atoms (on average) in the alkyl chain. The acid forms of other anionic detergent-active compounds, for example, fatty acid soaps and secondary alkane sulphonates, may also be used in the process of the invention if available in a suitable stable highly concentrated liquid form.

In the process of the invention, the bed particles are fluidised by means of an inert gas, preferably air. The air flow rate required for soda ash fluidisation is exceptionally low and it can be difficult to achieve uniform fluidisation. It is therefore advantageous for the fluidising gas to enter the bed via a distributor cap with relatively small holes, but blocking can then occur as the anionic surfactant precursor is sprayed on. A screw cap distributor plate has been found to be especially effective, but if desired a bubble cap plate or sieve plate, or any other suitable distributor plate known in the art, may be used.

DESCRIPTION OF DRAWING

The invention will now be described in more detail, by way of example only, with reference to the accompanying drawing which is a schematic vertical sectional view of apparatus suitable for carrying out the process of the invention.

Referring now to the accompanying drawing, an internal recirculating fluidised bed is indicated generally by the reference numeral 1. Solids are charged from a vibratory feeder (not shown) into the bed via a pneumatic charging line 2. Fluidising gas enters via a central draught tube 11 through air inlet pipe 3 as well as via a distributor plate 4, and leaves via a line 5 connected to a cyclone separator and a suction blower.

Two spray nozzles 6 and 7 are provided for the spray-in of liquids, fed respectively by liquid feed lines 8 and 9 and atomised by air lines (not shown). A line 10 is provided for discharge of product. The draught tube 11 is supported by support legs 12 and 13.

The apparatus described above may be used in the process of the invention as follows. Soda ash is fed by means of the vibratory feeder via the line 2 into the bed, and fluidised by air entering via the draught tube 3 and the distributor plate 4 and leaving via the line 5. Once uniform fluidisation has been achieved, alkylbenzene sulphonic acid is sprayed onto the bed through the spray nozzle 6, fed by the line 8. A heating jacket 14 maintains the alkylbenzene sulphonic acid in the line 8 at a temperature at which its viscosity is optimum for good spraying. Aqueous sodium silicate solution (40-45%) may if desired be sprayed onto the bed through the spray nozzle 7 fed by the line 9. Preferably the alkylbenzene sulphonic acid and sodium silicate solution are not sprayed simultaneously, because if they come into direct contact with each other insoluble siliceous species may be formed.

EXAMPLES

The invention will now be illustrated by the following non-limiting Examples.

Example 1

This Examples describes a batch process in accordance with the invention. The apparatus used was as described above with reference to the accompanying drawing, the bed diameter being 50 cm, and the spray nozzles being positioned approximately 30 cm above the top of the bed.

35 kg of soda ash (100 mesh) were charged into the bed and fluidised with air at a flow rate of 600 litre/min. Linear alkylbenzene sulphonic acid (LAS) was sprayed onto the bed at a rate of 4.5 kg/hour. As the LAS spray-on continued, it became necessary to increase the gas flow rate gradually from its initial value of 600 litre/min to about 1800 litre/min to maintain good fluidisation characteristics in the bed. The spray-in of LAS was continued until defluidisation and lumping in the bed occurred: the time taken for this to occur was about 90-120 minutes.

A sample removed after 90 minutes was a free-flowing granular solid containing about 20% by weight of linear alkylbenzene sulphonate and about 80% by weight of sodium carbonate. Its Rosin-Rammler average particle size was 512 μm.

This material is useful as a detergent powder, or component therefore, containing a high level of active detergent. If desired, various other particulate detergent ingredients, for example, bleaches, bleach activators, bleach precursors, enzymes, sodium sulphate or sodium silicate, could be admixed with this material to produce a more efficient and/or attractive detergent composition.

Example 2

This Example describes a continuous process in accordance with the invention. Again, the apparatus used was as described above with reference to the accompanying drawing.

The process was started up by feeding the granular material obtained in Example 1 (20% linear alkylbenzene

sulphonate, 80% sodium carbonate) into the bed and fluidising with an initial air flow rate of 1800 - 2000 litre/min. When the bed had reached a steady state, soda ash (100 mesh) was fed continuously into the bed and LAS sprayed on continuously at a ratio chosen to give a final product containing 25% by weight of alkylbenzene sulphonic acid and 75% by weight of sodium carbonate. The bed level was maintained by controlling the discharge manually. In contrast to the batch process of Example 1, it was not necessary to increase the air flow rate in order to maintain satisfactory fluidisation.

The product obtained was a free-flowing granular solid containing the desired amount of 25% by weight of linear alkylbenzene sulphonate. It will be noted that this level was higher that the highest level (20%) that could be achieved in batch operation. The Rosin-Rammler average particle size of the product was 720 µm.

Like the product of Example 1, this material is useful either as a simple alkaline detergent composition in its own right, or as a high-active-detergent component in a more complex product.

CLAIMS

1. A process for the preparation of a solid particulate material suitable for use as a detergent composition or a component thereof, which comprises fluidising a particulate material comprising at least one detergency builder salt in an internal recirculating fluidised bed, and spraying thereon liquid material comprising at least one detergent-active compound and/or at least one precursor thereof.

2. A process as claimed in claim 1, wherein the particulate material comprises at least one alkaline builder salt, and the liquid material sprayed on comprises at least one acid precursor of an anionic detergent-active compound.

3. A process as claimed in claim 2, wherein the alkaline builder salt comprises alkali metal carbonate.

4. A process as claimed in claim 3, wherein the alkali metal carbonate is sodium carbonate.

5. A process as claimed in claim 4, wherein the particulate material fluidised consists substantially wholly of sodium carbonate.

6. A process as claimed in any one of claims 2 to 5, wherein the anionic detergent-active compound is an alkylbenzene sulphonate.

7. A process as claimed in any one of claims 2 to 6, wherein the liquid acid precursor is sprayed on in an amount such that the resulting product contains from 5 to 40% by weight of the anionic detergent-active compound in alkali metal salt form.

8. A process as claimed in claim 7, wherein the liquid acid precursor is sprayed on in an amount such that the resulting product contains from 25 to 35% by weight of the anionic detergent-active compound in alkali metal salt form.

9. A process as claimed in any preceding claim, wherein the liquid material sprayed on comprises at least one nonionic detergent-active compound.

10. A process as claimed in any preceding claim, wherein the liquid material sprayed on comprises at least one anionic detergent-active compound in concentrated aqueous solution form.

11. A process as claimed in any preceding claim, wherein the liquid material sprayed on comprises concentrated aqueous sodium silicate solution.

12. A process as claimed in any preceding claim, wherein fluidising gas enters the fluidised bed via a screw cap distributor plate.

13. A process as claimed in claim 1, carried out using apparatus substantially as hereinbefore described with reference to, and as shown in, the accompanying drawing.

14. A process as claimed in claim 1, carried out substantially as hereinbefore described in Example 1 or Example 2.

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